

(12) UK Patent Application (19) GB (11) 2 235 697 (13) A

(43) Date of A publication 13.03.1991

(21) Application No 8631037.2

(22) Date of filing 30.12.1986

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(51) INT CL⁶
C22C 19/05

(52) UK CL (Edition K)
C7A AA23Y AA237 AA239 AA25Y AA260 AA263
AA266 AA299 AA30Y AA311 AA313 AA316 AA319
AA320 AA323 AA326 AA329 AA335 AA339 AA34Y
AA35X AA35Y AA350 AA37Y AA377 AA379 AA38X
AA381 AA383 AA385 AA387 AA389 AA409 AA41Y
AA416 AA418 AA42X AA422 AA425 AA428 AA459
AA48Y AA485 AA487 AA489 AA51Y AA523 AA525
AA527 AA529 AA53Y AA545 AA547 AA549 AA579
AA599 AA609 AA629 AA67X AA671 AA673 AA675
AA677 AA679 AA68X AA68Y AA681 AA682 AA683
AA685 AA686 AA687 AA689 AA69X AA693 AA695
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(58) Field of search
UK CL (Edition I) C7A
INT CL⁶ C22C

(54) Nickel-base superalloys

(57) Gas turbine engine efficiencies are increased through improvements in nickel-base superalloys used to make parts and components for gas turbine engines. These improved superalloys consist essentially of, in percentages by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf; 0-0.07C, 0-0.015B and 0-0.075Y, the balance being Ni and incidental impurities. They produce single crystal articles having improvements in temperature capability, based on stress rupture strength and low and high cycle fatigue properties. Because of their superior resistance to degradation by cyclic oxidation, and their resistance to hot corrosion, the superalloys possess a unique balance in mechanical and environmental properties.

Si?

Cr overlap
Co overlap
Al overlap
Ta overlap
Re broader
Hf overlap
C overlap
B broader
W overlap
Y broader

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DEC 1984

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IMPROVED AND PROPERTY-BALANCED NICKEL-BASE
SUPERALLOYS FOR PRODUCING SINGLE
CRYSTAL ARTICLES

The invention disclosed and claimed herein is related to the invention disclosed and claimed in co-assigned application Ser. No. 595,854 filed on April 2, 1984.

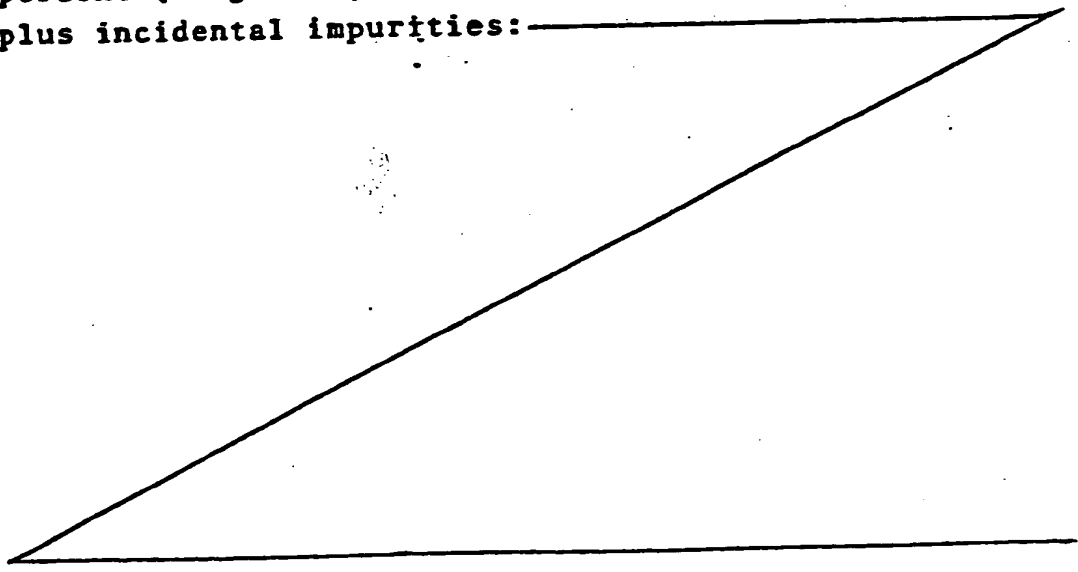
5 This invention pertains generally to nickel-base superalloys castable as single crystal articles of manufacture, which articles are especially useful as hot-section components of aircraft gas turbine engines, particularly rotating blades.

10 The efficiency of gas turbine engines depends significantly on the operating temperature of the various engine components with increased operating temperatures resulting in increased efficiencies. One means by which the operating temperature capability can
15 be increased is by casting the components which operate at the highest temperatures, e.g., turbine blades and vanes, with complex hollow passageways therein so that cooling air can be forced through the component and out through holes in the leading and trailing edges. Thus,
20 internal cooling is achieved by conduction and external cooling is achieved by film or boundary layer cooling.

The search for increased efficiencies has also led to the development of heat-resistant superalloys which can withstand increasingly high temperatures yet
25 maintain their basic material properties. Oftentimes,

the development of such superalloys has been done in conjunction with the design, development and manufacture of the aforementioned cast components having intricate air cooling passageways therein.

- 5 The present invention is directed to the achievement of increased efficiencies through further improvements in nickel-base superalloys. Accordingly, there is provided by the present invention nickel-base superalloys for producing single crystal articles
- 10 having a significant increase in temperature capability, based on stress rupture strength and low and high cycle fatigue properties, over single crystal articles made from current production nickel-base superalloys. Further, because of their superior
- 15 resistance to degradation by cyclic oxidation, and their resistance to hot corrosion, the superalloys of this invention possess a balance in mechanical and environmental properties which is unique and has not heretofore been obtained.

- 20 According to the present invention, superalloys suitable for making single-crystal castings comprise the elements shown in Table I below, by weight percent (weight %), with the balance being nickel (Ni) plus incidental impurities: _____
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-3-

TABLE I
ALLOY COMPOSITIONS
(weight %)

Elements		Base	Preferred	Most Preferred
5	Chromium (Cr):	5 - 10	6.75 - 7.25	7.0
	Cobalt (Co):	5 - 10	7.0 - 8.0	7.5
	Molybdenum (Mo):	0 - 2	1.3 - 1.7	1.5
	Tungsten (W):	3 - 10	4.75 - 5.25	5.0
	Tantalum (Ta):	3 - 8	6.3 - 6.7	6.5
10	Titanium (Ti):	0 - 2	0.02 max	0.0
	Aluminum (Al):	5 - 7	6.1 - 6.3	6.2
	Rhenium (Re):	0 - 6	2.75 - 3.25	3.0
	Hafnium (Hf):	0 - 0.50	0.12 - 0.18	0.15
	Carbon (C):	0 - 0.07	0.04 - 0.06	0.05
15	Boron (B):	0 - 0.015	0.003 - 0.005	0.004
	Yttrium (Y):	0 - 0.075	0.005 - 0.030	0.01

The invention also includes cast single-crystal articles, such as gas turbine engine turbine blades and vanes, made of an alloy having a composition falling within the foregoing range of compositions.

20 There are two basic directional solidification (DS) methods now well-known in the art by which single crystal castings may be made. They generally comprise either the use of a seed crystal or the use of a labyrinthine passage which serves to select a single
25 crystal of the alloy which grows to form the single crystal article ("choke" process).

In order to develop and test alloys of the invention, three series of 3000 gram heats of the alloys listed in Table II were vacuum induction melted
30 and cast into 1-1/2" dia. copper molds to form ingots. The ingots were subsequently remelted and cast into 1/2" x 2" x 4" single crystal slabs using the choke process, although the other previously mentioned process could have been used.

-4-

In a series of separate experiments, it was determined that yttrium retention in the single crystal slabs was about 30% of that present in the initial ingots. Hence, in preparing the series I, II, and III alloys shown in Table II, sufficient excess yttrium was added to the initially cast alloys so as to achieve the yttrium levels shown in Table II taking into account the 30% retention factor.

The series I alloys were designed to evaluate the interactions between tungsten, molybdenum and rhenium as gamma (γ) matrix alloying elements. The series II alloys were designed somewhat independently from series I in order to accommodate additional variables. Aluminum was maintained at a high level and titanium and tantalum were varied to accomplish a range of gamma prime (γ') levels up to about 63 volume percent and chromium was reduced in order to permit the increased γ' contents. Since it was determined that the 8% Cr series I alloys as a group were less stable than the series II alloys, the base Cr level was reduced from the 8% in series I to 7% to achieve better stability. Co was varied in alloys 812-814 to evaluate the effect of Co on stability.

The series III alloys were based on evaluations of the series I and II alloys. From series II, the upper limit in γ' content, based on γ' solutioning, was about 60 volume percent. Alloys 824-826 were based on alloy 820 which had 5.5% Re and high strength, but was unstable. Thus, the Re content was reduced to achieve stability. Alloys 827-829 were based on alloy 821 (0% Ti), but in which W and Re were varied. Alloys 830-833 were based on alloy 800 (1.5% Ti), but in which Re, W and Mo were varied. Alloys 834 and 835 contained increased Al at the expense of Ta and Ti. In all the series III alloys, the Co content was

maintained at 10%, based on the evaluation of alloys 812-814 in series II.

The series I, II and III alloys were evaluated for stress rupture strength and the results of the 5 tests are set forth in Table III. Prior to testing, the alloys, except for the "R" series noted in Table III, were heat treated as 1/2" thick single crystal slabs according to the following schedule:

10 solutionizing at 2350 - 2400°F for two hours to achieve solutioning of at least 95% of the γ' phase followed by an intermediate age at 1975°F for 4 hrs. and a final age at 1650°F for 16 hrs.

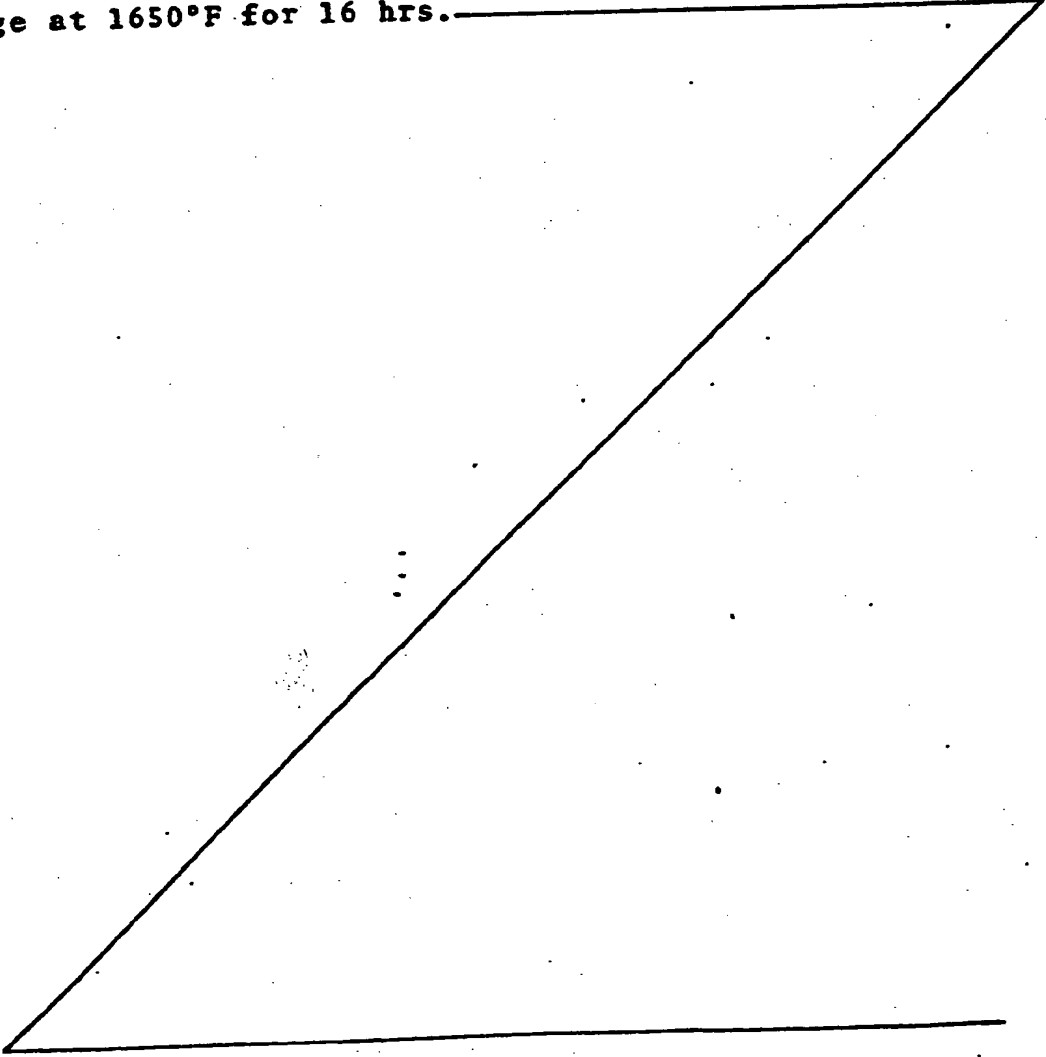


TABLE II
(single crystal analyses)

Series I

Alloy #	Cr	Co	Mo	W	Ta	Ti	Al	Re	Hf	B	C	Y
800	8	7.5	1.5	4.0	5	1.5	5.8	3.0	0.15	0.004	0.05	0
801	8	7.5	0.5	5.9	5	1.5	5.75	3.0	0.15	0.004	0.05	0
802	8	7.5	0.0	5.9	5	1.5	5.75	3.0	0.15	0.004	0.05	0.015
803	8	7.5	0.0	4.0	5	1.5	5.75	4.5	0.15	0.004	0.05	0
804	8	7.5	0.0	2.0	5	1.5	5.75	6.0	0.15	0.004	0.05	0
805	8	7.5	3.65	0.0	5	1.5	5.9	3.1	0.15	0.004	0.05	0
806	8	7.5	3.0	0.0	5	1.5	5.8	4.5	0.15	0.004	0.05	0
807	8	7.5	1.5	3.0	6	1.0	6.0	3.0	0.15	0.004	0.05	0
808	8	7.5	1.5	3.0	6	0.0	6.5	3.0	0.15	0.004	0.05	0.015
809	8	7.5	0.0	4.0	6	0.0	6.4	4.5	0.15	0.004	0.05	0
810	8	7.5	0.0	2.0	6	0.0	6.4	6.0	0.15	0.004	0.05	0
811	8	7.5	3.0	0.0	6	0.0	6.4	4.5	0.15	0.004	0.05	0

10

5

10

TABLE II
(single crystal analyses)

Series II

Alloy #	Cr	Co	Mo	W	Ta	Ti	Al	Re	Hf	C	B	Y
812	7	5.0	1.5	3	6.0	1.0	6.0	3.0	0.15	0.05	0.004	0.015
813	7	7.5	1.5	3	6.0	1.0	6.0	3.0	0.15	0.05	0.004	0.015
814	7	10.0	1.5	3	6.0	1.0	6.0	3.0	0.15	0.05	0.004	0.015
815	5	7.5	1.5	3	7.5	0.5	6.5	3.0	0.15	0.05	0.004	0.015
816	5	7.5	1.5	3	8.0	0.5	6.5	3.0	0.15	0.05	0.004	0.015
817	5	7.5	0.5	3	8.0	1.0	6.5	3.0	0.15	0.05	0.004	0.015
818	5	7.5	1.5	3	7.0	0.5	6.5	3.5	0.15	0.05	0.004	0.015
819	5	7.5	1.5	3	7.0	0.5	6.5	4.5	0.15	0.05	0.004	0.015
820	5	7.5	1.5	3	7.0	0.0	6.5	5.5	0.15	0.05	0.004	0.015
821	7	7.5	1.5	5	6.5	0.0	6.2	3.0	0.15	0.05	0.004	0.015
822	6	7.5	1.5	5	6.5	0.0	6.2	3.0	0.15	0.05	0.004	0.015
823	5	7.5	1.5	5	6.5	0.0	6.2	3.0	0.15	0.05	0.004	0.015

TABLE II
(single crystal analyses)

Series III

Alloy #	Cr	Co	Mo	W	Ta	Ti	Al	Re	Hf	B	C	Y
824	5.0	10.0	1.5	6.5	7.0	0	6.5	2.0	0.15	0.004	0.05	0.015
825	5.0	10.0	1.5	5.5	7.0	0	6.5	3.0	0.15	0.004	0.05	0.015
826	5.0	10.0	1.5	4.0	7.0	0	6.5	4.0	0.15	0.004	0.05	0.015
827	7.0	10.0	1.5	5.0	6.5	0	6.2	3.0	0.15	0.004	0.05	0.015
828	7.0	10.0	1.5	6.0	6.5	0	6.2	2.5	0.15	0.004	0.05	0.015
829	7.0	10.0	1.5	7.0	6.5	0	6.2	2.0	0.15	0.004	0.05	0.015
830	7.0	10.0	1.5	5.0	5.0	1.5	5.8	3.0	0.15	0.004	0.05	0.015
831	7.0	10.0	1.5	6.0	5.0	1.5	5.8	2.0	0.15	0.004	0.05	0.015
832	7.0	10.0	1.5	7.0	5.0	1.5	5.8	1.0	0.15	0.004	0.05	0.015
833	7.0	10.0	2.5	4.0	5.0	1.5	5.8	2.0	0.15	0.004	0.05	0.015
834	6.0	10.0	1.5	5.5	4.0	0	7.0	3.0	0.15	0.004	0.05	0.015
835	7.0	10.0	1.5	4.0	2.0	0	7.5	3.0	0.15	0.004	0.05	0.015

TABLE III
Stress Rupture
 (parallel to single crystal growth direction)

ALLOY	AC01	LIFE (HRS)			
		1600°F/ 80 Ksi	1800°F/ 40 Ksi	2000°F/ 20 Ksi	2100°F/ 13 Ksi
5					
800		94.5	68.5	184.5	90.4
801		87.0	44.5	45.5	29.1
802		86.8	63.1	90.0	-
803		66.7	67.1	70.1	82.9
10		54.2	103.0	52.4	-
804		56.3	56.3	55.2	30.0
805		85.6	43.8	57.4	22.9
806		75.7	60.1	100.3	66.4
807		55.6	53.5	31.6	39.2
808		22.0	69.6	46.0	25.3
15	N	62.2	64.7	35.4	10.1
809		101.9	161.8	44.1	8.2
810		35.1	49.1	30.2	53.5
811		53.8	51.4	27.0	52.2
812		57.9	63.7	42.8	47.1
20		76.5	65.4	64.8	143.2
813		103.9	83.6	47.6	121.4
814		24.8	55.5	42.3	55.5
815		84.3	85.6	68.0	113.3
816	N	147.4	115.5	100.6	264.3
817		257.2	158.7	153.7	220.1
25		114.3	80.4	98.4	74.3
818		64.2	70.3	43.1	96.9
819		22.3	48.7	-	46.3
820					
821					
822					
823					

(continued)

-10-

TABLE III - continued

Stress Rupture

ALLOY	ACO ¹	LIFE (HRS)		
		1600°F/ 80 Ksi	1800°F/ 40 Ksi	2000°F/ 20 Ksi
5 824		97.1	91.9	
	R		118.8	67.7
825		74.0	94.7	
	R		128.4	82.3
826		113.1	119.8	
10 827	N	6.7	135.6	122.1
	R		76.2	
828		119.1	108.4	70.0
	R		72.7	
15 829		110	95.7	119.0
	R		72.7	
830	N	59.8	90.2	88.0
	R		126.5	
831		92.7	162.2	147.8
20 832		90.1	68.9	
	R		82.1	137.6
833		96.5	58.5	
	R		85.4	107.3
25 834		119.2	51.6	
	R		69.1	132.7
835	N	43.5	80.7	
	R		105.7	69.1
			55.3	
			67.4	27.9

1 ACO= Acceptable Crystallographic Orientation =
 30 single crystal growth orientation within 15° of the
 [001] zone axis; N = no, otherwise yes

The series III alloys were initially tested at
 1600°F/80 ksi and 1800°F/40 ksi. Based on other tests,
 such as those reported in Table VII, additional test
 35 specimens were resolutioned at 2390°F for two hours,
 given a more rapid cool and aged at 2050°F/4 hours +
 1650°F/4 hours, the "R" treatment listed in Table III,

-11-

and stress rupture tested at 1800°F/40 ksi and 2000°F/20 ksi. The reheat treatment resulted in an average increase in rupture life at 1800°F/40 ksi of about 30%. At the critical parameter of 1800°F/40 ksi for gas turbine engine applications, it is expected that the series I and II alloys would also exhibit a 30% increase in life when given the "R" treatment.

Other experiments have shown that cooling rates from the solutionizing temperature to 2000°F in the range of 100-600°F/min have only a slight effect on the stress rupture properties of the alloys of the invention with higher rates tending to improve the life at 1800°F/40 ksi slightly. The data are shown in Table IV.

15

TABLE IV

	<u>Cooling Rate</u> <u>°F/Min</u>	<u>Stress Rupture Life, Hours</u>	
		<u>1800°F/40 ksi</u>	<u>2000°F/20 ksi</u>
	600	91	107
	300	85	123
20	100-150	75	120
	Average of all prior data (vari- ous cooling rates)	79	100

Thus, for the superalloys of the invention, the presently preferred heat treatment is as follows: solutionize in a temperature range sufficient to achieve solutioning of at least 95% of the γ' phase, preferably 2385-2395°F, for 2 hrs., cool to 2000°F at 100°F/min. minimum, furnace cool to 1200°F in 60 min. or less and thereafter cool to room temperature; heat

-12-

to $2050 \pm 25^\circ\text{F}$ for 4 hrs., furnace cool to below 1200°F in 6 min. or less and thereafter to room temperature; and heat to $1650 \pm 25^\circ\text{F}$ for 4 hrs. and thereafter furnace cool to room temperature. All heat treatment steps are performed in vacuum or an inert atmosphere, and in lieu of the steps calling for cooling to room temperature the treatment may proceed directly to the next heating step.

The stress rupture data from the series I, II, and III alloys indicates that about 5% Re provides the highest rupture strength at $1800^\circ\text{F}/40$ ksi. The data also show, when rupture life is graphed as a function of rhenium content at constant tungsten contents, that high rupture life at $1800^\circ\text{F}/40$ ksi can be obtained with rhenium plus tungsten levels in the (3Re + 7W) to (5Re + 3W) ranges. In the most preferred embodiment, Alloy 821, the presently preferred (Re + W) combination is (3Re + 5W) due to the present relative costs of rhenium and tungsten.

All the alloys were evaluated for microstructural stability. Specimens were heat treated by solutionizing at $2375\text{--}2400^\circ\text{F}/2$ hrs. and aging at $1975^\circ\text{F}/4$ hrs. and at $1650^\circ\text{F}/16$ hrs. Thereafter, different sets of specimens were heated for 1000 hrs. at 1800°F and for 1000 hrs. at 2000°F . After preparation, including etching with diluted Murakami's electrolyte, the specimens were examined metallographically and the relative amount of topologically close packed phase (TCP) was determined visually. The series II alloys, except alloys 818 and 819, showed either no TCP precipitation or only traces of precipitation (821) and, as a group, were less prone to microstructural instability than the series III alloys and much less prone than the series I alloys at both 1800°F and 2000°F .

Table V presents the results of cyclic oxidation tests on uncoated 1/4" dia. x 3" long pin specimens conducted at 2150°F using a natural gas flame at Mach 1 gas velocity. The specimens were rotated for
5 uniform exposure and cycled out of the flame once per hour to cool the specimens to room temperature. External metal loss was measured on a section cut transverse to the length dimension of the specimen. Metal loss per side was found by dividing the
10 difference between the pin diameter before and after test by two. The data in the table are the average of two such measurements at 90° to each other across the diameter of the specimen.

The two series I alloys that contained yttrium
15 (802 and 808) had exceptional oxidation resistance. The series II alloys, all of which were yttrium-bearing, exhibited no metal loss after 200 hours of high velocity oxidation (Mach I) at 2150°F and only 2-3 mils γ' depletion, demonstrating that a
20 synergistic Y + Hf effect was operating. These data also demonstrate that Re improves the oxidation resistance or at least is less detrimental than W which it has replaced in the alloys and, from metallographic studies, also results in improved γ' stability.

-14-

TABLE V

Oxidation, 2150°F, Mach 1.0

Alloy	Time (hrs)	Metal Loss (mils/side)					γ' Depletion (mils/side)
		18.6	62.6	127.6	169.6	214.6	
5							
	800	1.0	1.8	4.8	6.8	9.3	6-12
	801	0.8	1.5	4.8	8.0	9.8	4-8
	802	0	0	0.3	0	0.3	2-3
	803	0.8	1.5	2.8	4.0	5.8	8-10
10	804	0.8	1.3	1.5	4.0	5.3	8-12
	805	1.0	1.5	10.3	7.5	9.5	8-10
	806	0.8	1.8	4.8	6.3	9.8	8-10
	807	1.0	2.0	4.5	6.3	8.0	6-8
	808	0.3	0	0.3	0.3	0.5	2-3
15	809	1.0	1.8	2.8	2.0	4.3	10-14
	810	1.0	1.8	2.8	3.5	4.0	10-16
	811	1.3	2.5	3.0	4.5	5.5	12-16
	812	0	0	0	0	0	1-2
	813	0	0	0	0	0	1-2
20	814	0	0	0	0	0	1-2
	815	0	0	0	0	0	1
	816	0	0	0	0	0	1
	817	0	0	0	0	0	2
	818	0	0	0	0	0	2
25	819	0	0	0	0	0	2
	820	0	0	0	0	0	1-2
	821	0	0	0	0	0	1-2
	822	0	0	0	0	0	1-2
	823	0	0	0	0	0	1-2
30	R125	-	-	-	-	80	-
	R80	-	-	-	-	90	-
	MA754	-	-	-	-	12	-

-15-

The hot corrosion resistance of the alloys of the invention was evaluated alongside three alloys used to produce production turbine blades, Rene' 125, B1900, and MM200(Hf), in tests wherein specimens of the alloys were exposed to a JP-5 fuel-fired flame at 1600°F with a nominal 1 ppm salt added to the combustion products. The test was first run at ~1 ppm for 1040 hrs., and then at ~2 ppm, for 578 hrs. The chemical determination of NaCl on calibration pins at every 200 hours indicated that the salt level was between 0 and 1 ppm during the first 1000 hours, between 1 and 2 ppm during the next 300-400 hours and above 2 ppm during the remaining 300 hours. The following conclusions were drawn from these hot corrosion tests: 1) B1900 was least resistant to hot corrosion at all salt levels, 2) MM200(Hf) was the next least resistant alloy at all salt levels, 3) the alloys of the invention, especially alloy 821, and Rene' 125 exhibit similar hot corrosion behavior, with the alloys of the invention being slightly less resistant than Rene' 125, and 4) as is the case for Rene' 125 and other alloys, the alloys of the invention appear to be sensitive to salt level in the corrosion test with increased salt level resulting in poorer corrosion resistance. Thus, the difference between B1900, MM200(Hf), Rene' 125, and the alloys of the invention narrows at high salt levels. These results are consistent with prior experience and indicate that the hot corrosion resistance of the alloys of the invention will be adequate for applications where Rene' 125 equivalency is required.

Alloy 821 was scaled up as a 300 lb master heat having the composition given in Table VI. No yttrium was added to the master heat; rather, yttrium was added when the master heat material was remelted and molten

-16-

prior to DS'ing to produce single crystal slabs and turbine blades. For the test specimens used to obtain the data of Tables VII, VIII, IX, and X, yttrium in the amount of 400 ppm was added. Stress rupture strength data for alloy 821 from the 300 lb master heat and the 12 lb. laboratory heat are presented in Table IX.

TABLE VI300 Lb Alloy 821 Master Heat

Cr	6.79	Ti	0
Co	7.30	Re	2.95
Mo	1.48	Hf	0.17
W	4.95	C	0.05
Ta	6.40	B	0.004
Al	6.15	Y	0

10

TABLE VII

Stress Rupture Data

Heat	H. Treat	Temp (°F)	Stress (ksi)	Life (Hrs)	El (%)	RA (%)
5	12 lb Lab. Ht.	2390/2 + 1975/4	1600	80	114.3	
		+ 1650/16	1800	40	80.4	
		H.T. as slabs	2000	20	98.4	7.7 43.1
			2100	13	74.3	16.8 6.8
10	300 lb Master Ht. Alloy 821	2390/2 + 1975/4	1400	130	1.9	19.5 26.9
		+ 1650/16	1400	110	351.6	14.8 24.4
		H.T. as slabs	1600	80	155.4	20.1 26.8
			1800	40	72.7	39.4 29.9
			1800	40	75.8	20.6 33.2
			1800	35	227.8	17.5 27.3
			1800	30	509.2	16.8 28.7
			1900	25	120.2	10.1 23.4
			1900	22	357.2	13.9 28.6
			2000	20	81.3	13.6 38.5
20	300 lb Alloy 821		2000	17.5	391.9	13.1 23.3
			2100	13	80.5	3.4 48.6
		Reheat treated*	1600	80	115.8	19.0 25.0
		+ 1900/4 age +	1800	40	68.4	17.0 30.5
25		1650/4 age	2000	20	82.7	13.9 35.2
		Reheat treated*	1600	80	155.2	19.0 26.2
		+ 1975/4 age +	1800	40	85.2	25.5 39.0
		1650/4 age	2000	20	101.2	14.7 34.4
30		Reheat treated*	1600	80	160.0	18.9 27.5
		+ 2050/4 age +	1800	40	103.8	18.1 28.3
		1650/4 age	2000	20	125.7	11.6 40.3
		Reheat treated*	1600	80	139.9	19.3 24.0
35		+ 2125/1 age +	1800	40	97.4	23.2 28.6
		+ 1975/4 (coating	2000	20	126.9	12.8 32.9
		simulation) +				
		1650/4 age				
40		Reheat treated*	1600	80	131.0	17.8 24.7
		+ 2200/1 age +	1800	40	90.5	20.6 29.8
		+ 1975/4 (coating	2000	20	97.2	12.4 31.1
		simulation) +				
40		1650/4 age				

* All resolutioned in test specimen form at 2390°F/2 hr + fast cool to 2000° F.

-18-

5 Tensile strength, low cycle fatigue and high cycle fatigue tests were performed on single crystal material from the 300 lb heat of alloy 821 solutioned at 2390°F/2 hrs. and aged at 1975°F/4 hrs. and 1650°F/16 hrs., with the results shown in Tables VIII, IX, and X, respectively, where UTS is ultimate tensile strength; YS is yield strength at 0.2% strain offset; El is elongation; and RA is reduction in area.

TABLE VIII

Tensile Data

(Master Heat Alloy 821)

Temp (°F)	UTS (Ksi)	0.2% YS (Ksi)	0.02% YS (Ksi)	El (%)	RA (%)
1000	128.6	113.4	110.7	11.6	18.9
15 1200	129.6	112.4	106.5	14.2	19.9
1400	142.8	112.8	102.6	9.9	13.3
1600	143.3	129.4	103.5	18.0	30.8
1800	110.1	94.7	71.9	10.0	28.1
2000	64.1	51.2	39.2	19.1	21.6

TABLE IX

Low Cycle Fatigue

(Master Heat Alloy 821)

Alternating Pseudostress (ksi) ¹	Cycles to Failure N _f
21	4.9 x 10 ³
31	2.3 x 10 ³
37	2.5 x 10 ³

¹ 2 min. compressive strain hold, 2000°F

TABLE X
 High Cycle Fatigue¹
 (Master Heat Alloy 821)

	Alternating Stress (ksi)	Cycles to Failure N _f
5	10	9.6 x 10 ⁶
	11	4.4 x 10 ⁶
	13	1.4 x 10 ⁶
	15	0.5 x 10 ⁶

¹ 2050°F; A = 0.67, 30 Hz

10 As discussed at greater length in co-pending
 co-assigned application Ser. No. 595,854, the
 superalloys of this invention break with the
 long-standing wisdom of the single crystal superalloy
 arts that grain boundary strengthening elements such as
 15 B, Zr and C are to be avoided, i.e., kept to the lowest
 levels possible consistent with commercial melting and
 alloying practice and technology. One general
 reason given for restricting such elements is to
 increase the incipient melting temperature in relation
 20 to the γ' solvus temperature thus permitting
 solutionizing heat treatments to be performed at
 temperatures where complete solutionizing of the γ'
 phase is possible in reasonable times without causing
 localized melting of solute-rich regions. Another is
 25 to minimize or preclude the formation of deleterious
 TCP phases.

As noted in the Ser. No. 595,854 application,
 single crystal articles are not necessarily wholly of a

single crystal as there may be present therein grain boundaries referred to as low angle grain boundaries wherein the crystallographic mismatch across the boundary is generally accepted to be less than about 5
5 to 6 degrees. Low angle grain boundaries are to be distinguished from high angle grain boundaries which are generally regarded as boundaries between adjacent grains whose crystallographic orientation differs by more than about 5-6 degrees. High angle grain
10 boundaries are regions of high surface energy, i.e., on the order of several hundreds of ergs/cm², and of such high random misfit that the structure cannot easily be described or modelled.

As also noted therein, the discovery that
15 small, but controlled, amounts of such previously prohibited elements can be tolerated resulted in the single crystal superalloys of the Ser. No. 595,854 application which have improved tolerance to low angle grain boundaries, i.e., have greater grain boundary
20 strength than the state-of-the-art single crystal superalloys. As one result of this increased grain boundary strength, grain boundary mismatches far greater than the 6° limit for prior art single crystal superalloy articles can be tolerated in single crystal
25 articles made from the nickel-base superalloys of that invention. This translates, for example, into better in-service reliability, lower inspection costs and higher yields as grain boundaries over a broader range can be accepted by the usual inspection techniques.
30 The novel features of that invention have been embodied in the novel superalloys of the present invention; thus, the superalloys of the present invention also exhibit improved tolerance to low angle grain boundaries and also have the above-described benefits.

The superalloys of this invention are also alloyed with yttrium which renders them more highly reactive with respect to ceramic molds and cores used in the investment casting process than nickel-base superalloys not alloyed with yttrium. Ceramic/metal instability is controlled by the bulk thermodynamic condition of the system. The more negative the free energy of formation, ΔG°_f , the greater the affinity for oxygen. It has been found that the free energy of formation for oxides becomes more negative as more reactive elements, such as yttrium, are added resulting in a greater potential for metal/ceramic reaction than when typical SiO_2 and ZrO_2 ceramic mold and core systems are used. Based on thermodynamic considerations and the work reported in U.S. Department of the Air Force publication AFML-TR-77-211, "Development of Advanced Core and Mold Materials for Directional Solidification of Eutectics" (1977), alumina is less reactive and is, therefore, a preferred material for molds, cores and face coats when casting superalloys containing reactive elements.

It has also been found that melt/mold and core interactions are decreased, the retention of yttrium increased and the uniformity of yttrium distribution improved by the use of low investment casting parameters and temperatures. This translates to the use of the lowest possible superheat and mold preheat and a high withdrawal rate in the casting of the single crystal articles of this invention

Several uncored small turbine blades were investment cast using alloy 821 material from the previously mentioned 300 lb scale-up master heat. Those blades measured about 1.5" from tip to root with a span of approximately 0.75". Blade tip to platform distance was 1". As noted earlier, yttrium was added

-22-

to the master heat material while molten and prior to DS'ing -- in this case the amount was 2000 ppm. In general, most blades exhibited acceptable crystal structure and, as shown in Table XI, those cast using low casting parameters had better yttrium retention. Also, it appeared that surface to volume ratio influences yttrium retention; as the ratio increases, the yttrium retention decreases. This is illustrated by comparison of yttrium retention at the leading and trailing edges; the surface to volume ratio is lower in the leading edge compared to the trailing edge, and the yttrium retention in the leading edge is consistently higher than at the trailing edge.

TABLE XI

		Yttrium Content (ppm)				
15		Airfoil Tip		Airfoil Near Platform		Blade Root
	<u>Casting Condition</u>	<u>LE</u> (1)	<u>TE</u> (2)	<u>LE</u>	<u>TE</u>	<u>ROOT</u> ³
20	Low	130	100	160	100	130
	Superheat	90	60	80	50	160
		190	120	190	150	190
		170	90	180	150	200
		410	330	470	360	380
		310	120	270	160	280
25	High	80	60	120	70	100
	Superheat	80	80	100	70	130
		100	90	90	150	100
		80	60	100	100	100
		130	150	190	150	120
		170	200	240	210	170
30						

1 LE = leading edge

2 TE = trailing edge

3 ROOT = root, center

-23-

Additional single crystal investment castings of large solid turbine blades (4-3/4" tip-to-root) and small and large turbine blades having cores therein to define serpentine passageways for the provision of cooling air were also made. The large solid turbine blades required late yttrium additions of up to 2400 ppm in order to obtain yttrium distributions within the desired 50-300 ppm level. Similar such levels, coupled with the use of low investment casting parameters, were required to obtain acceptable yttrium levels in the cored blades. As was the case with the uncored small turbine blades, the effect of surface to volume ratio was evident; the leading edge retained higher yttrium levels compared to the trailing edge.

Although the present invention has been described in connection with specific examples, it will be understood by those skilled in the art that the present invention is capable of variations and modifications within the scope of the invention as represented by the appended claims.

CLAIMS

What is claimed is:

1. A nickel-base superalloy consisting essentially of, in percentages by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and incidental impurities.
- 5 2. The superalloy of claim 1 consisting essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max. Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf, 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.
- 5 3. The superalloy of claim 2 consisting essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf, 0.05 C, 0.004 B, and 0.01 Y, the balance being nickel and incidental impurities.
- 5 4. A nickel-base superalloy for producing single crystal articles consisting essentially of, in percentages by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and incidental impurities.
- 5 5. The superalloy of claim 4 consisting essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf, 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.
- 5 6. The superalloy of claim 5 consisting essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf,

0.05 C, 0.004 B, and 0.01 Y, the balance being nickel
5 and incidental impurities.

7. The article of claim 4 which is an airfoil member for a gas turbine engine.

8. A nickel-base superalloy for producing single crystal articles having improved tolerance to low angle grain boundaries consisting essentially of, in percentage by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10
5 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and incidental impurities.

9. The superalloy of claim 8 consisting essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max. Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf,
5 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.

10. The superalloy of claim 9 consisting essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf, 0.05 C, 0.004 B, and 0.01 Y, the balance being nickel and
5 incidental impurities.

11. The article of claim 8 which is an airfoil member for a gas turbine engine.

12. A heat treatment conducted in a vacuum or inert atmosphere comprising the steps of:

a) selecting a single crystal article having a composition consisting
5 essentially of, in percentages by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and
10 incidental impurities;

-26-

- b) heating for 2 hours in a temperature range sufficient to achieve solutioning of at least about 95% of the γ' phase;
c) cooling to 2000°F at 100°F/minute minimum and then to 1200°F in 60 minutes or less;
d) heating to 2050 \pm 25°F for 4 hours;
e) cooling to below 1200°F in 6 minutes or less; and
f) heating to 1650 \pm 25°F for 4 hours.

13. The heat treatment of claim 12 wherein the composition of the article consists essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max. Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf, 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.

14. The heat treatment of claim 12 wherein the composition of the article consists essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf, 0.05 C, 0.004 B, and 0.01 Y, the balance being nickel and incidental impurities.

15. The heat treatment of claim 12 wherein the temperature range in b) is from about 2385 to 2395° F.

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9/10/85

Amendments to the claims have been filed as follows

1. A nickel-base superalloy consisting essentially of, in percentages by weight, 5-10 Cr, 5-10Co, 0-2 Mo, 3-10 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and incidental impurities.
5
2. The superalloy of claim 1 consisting essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max. Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf, 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.
10
3. The superalloy of claim 2 consisting essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf, 0.05 C, 0.004 B, and 0.01 Y, the balance being nickel and incidental impurities.
15
4. A single crystal article produced from a nickel-base superalloy having a composition according to any one of the preceding claims.
20
5. An article according to claim 4 which is an airfoil member for a gas turbine engine.
25
6. A heat treatment conducted in a vacuum or inert atmosphere comprising the steps of:
30
 - (a) selecting a single crystal article having a composition consisting essentially of, in percentages by weight, 5-10 Cr, 5-10 Co, 0-2 Mo, 3-10 W, 3-8 Ta, 0-2 Ti, 5-7 Al, 0-6 Re, 0-0.5 Hf, 0-0.07 C, 0-0.015 B, and 0-0.075 Y, the balance being nickel and incidental impurities;
35

- b) heating for 2 hours in a temperature range sufficient to achieve solutioning of at least 95% of the γ' phase;
- c) cooling to 2000°F at 100°F/minute minimum and then to 1200°F in 60 minutes or less;
- d) heating to 2050 \pm 25°F for 4 hours;
- e) cooling to below 1200°F in 6 minutes or less; and
- f) heating to 1650 \pm 25°F for 4 hours.

7. The heat treatment of claim 6 wherein the composition of the article consists essentially of, in percentages by weight, 6.75-7.25 Cr, 7.0-8.0 Co, 1.3-1.7 Mo, 4.75-5.25 W, 6.3-6.7 Ta, 0.02 max. Ti, 6.1-6.3 Al, 2.75-3.25 Re, 0.12-0.18 Hf, 0.04-0.06 C, 0.003-0.005 B, and 0.005-0.030 Y, the balance being nickel and incidental impurities.

8. The heat treatment of claim 6 wherein the composition of the article consists essentially of, in percentages by weight, 7 Cr, 7.5 Co, 1.5 Mo, 5 W, 6.5 Ta, 0 Ti, 6.2 Al, 3 Re, 0.15 Hf, 0.05 C, 0.004 B, and 0.01 Y, the balance being nickel and incidental impurities.

9. The heat treatment of claim 6 wherein the temperature range in b) is from about 2385 to 2395°F.

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